



DRIFT SCALE TEST GAS TRACER TESTING IN HYDROLOGY BOREHOLES USING THE BALZER'S MASS SPECTROMETER

PROCEDURE ID: YMP-LBNL-TIP/TT 2.0

REV. 0, MOD. 0

EFFECTIVE: 09/30/98

1. PURPOSE

This Technical Implementing Procedure (TIP) describes a method for Gas Tracer Testing in the Drift Scale Test (DST) Area of the Yucca Mountain Site Characterization Project (YMP) at Ernest Orlando Lawrence Berkeley National Laboratory (LBNL).

The objective of Gas Tracer Testing in the DST Area is to estimate the effective gas-filled porosity of the formation as thermal testing is being conducted. To assure the accuracy, validity, and applicability of the method used to perform Gas Tracer Testing in the DST area, this procedure directs LBNL personnel and contractors performing the described activity.

This procedure describes the components of the work involved in gas tracer testing in the DST area. It also describes the methods to be used for calibration, operation, and performance verification of any equipment, if needed. In addition, it defines requirements for data acceptance, documentation, and control.

2. SCOPE

This procedure applies to all LBNL personnel or contractor personnel following LBNL procedures who may conduct Gas Tracer Testing in the DST Hydrology Boreholes using the Omnistar Mass Spectrometer (MS). The hydrology boreholes consist of three clusters of boreholes in the DST Area commonly referred to as Boreholes 57 to 61, Boreholes 74-78, and Boreholes 185 and 186. The Omnistar is a quadrupole MS that uses a Faraday Cup and Channeltron Detector, and is produced by Balzer's.

For all technical activities, data collected using this procedure and any equipment calibrations or recalibrations that may be required shall be in accordance with this TIP and in full compliance with YMP-LBNL-QIP-12.0, *Control and Calibration of Measuring and Testing Equipment*.

If this procedure cannot be implemented as written, YMP-LBNL personnel shall notify the responsible Principal Investigator (PI) or designee. If it is determined that a portion of the work cannot be accomplished as described in this TIP, or would produce undesirable results, that portion of the work shall be stopped and not resumed until this procedure is modified per YMP-LBNL-QIP-5.2, *Preparing Quality & Technical Implementing Procedures*.

If the responsible PI or designee determines that a modification or a revision to the TIP would cause an unreasonable delay in proceeding with the task, then an

expedited change to the procedure, including documentation of deviation from the approved procedure, can be made according to YMP-LBNL-QIP-5.2. Such changes are subject to review, usually after the task has proceeded, and thus work performed under TIPs with expedited changes is done at risk of future invalidation.

Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used. When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

3. PROCEDURE

3.1 Gas Tracer Testing Components

Gas tracer testing utilizes two distinct flow control systems: the Tracer Injection system, which is a modification of the Flow Control System used in constant mass flux injection testing and a Gas Withdrawal System. There is also a data acquisition system, which is identical to the hardware described in YMP-LBNL-TIP/TT 1.0, *Drift Scale Test Constant Mass Flux Air-Permeability Testing in Hydrology Boreholes*, but utilizes modified software.

3.1.1 Flow Control System

Ambient air supplied through the tunnel compressed air system is first filtered, dehumidified using a regenerative twin-tower desiccator, and filtered again before being sent through mass flow controllers. Attachment 1 is a piping diagram for the injection gas control system. Four Sierra Instruments mass flow controllers (MFCs), with full flow ranges of 1 Standard Liter Per Minute (SLPM), 10 SLPM, 100 SLPM, and 500 SLPM, which are connected in parallel, are selectively used to control the precise amount of gas being introduced into a borehole interval. The outlet of each mass flow controller has a pneumatically controlled valve to ensure positive shut off when the mass flow controller is not in use. Downstream of the mass flow controller manifold is an array selection manifold. The array selection manifold directs the gas flow to one of the three arrays of hydrology boreholes, Boreholes 57 to 61, Boreholes 74 to 78, or Boreholes 185 and 186. Another manifold is located near the collars of each borehole that directs the gas flow to the isolated zones (packed-off intervals) within the borehole.

3.1.2 Tracer Injection System

The Gas Tracer Injection System, which is an expansion of the Flow Control System utilized for constant mass flux injection testing, is designed to deliver a known concentration of traced gas for a precisely controlled duration of time at a known flow rate. Attachment 1 shows the layout of the Tracer Injection System. Depending upon the concentration requirements for the trace gas stream, the trace gas MFC may use either a 1 SLPM MFC or a 100 standard cubic centimeter per minute (SCCM) MFC. The maximum permissible concentration of SF₆, Neon or Xenon to be injected is 1000 PPM. The tracer injection system utilizes a gas regulator and MFC to control the flow of tracer that will be mixed in with the ambient air controlled by the Flow Control System.

3.1.3 Gas Withdrawal System

The Gas withdrawal system is designed to withdraw a controlled gas stream from a packed-off interval in the hydrology boreholes. A small portion of the stream is diverted into a Quadrupole MS for analysis of tracer concentration. A plumbing diagram for the Gas Withdrawal System is shown in Attachment 2. Immediately downstream from the sample collection point is a GAST diaphragm pump. The outlet of the GAST pump is divided into two gas streams: a rapid stream entering a refrigerated air dryer and controlled by a 0-100 SLPM MFC, and a 50 to 100 SCCM stream, controlled by a variable speed peristaltic pump used for gas analysis. The variable speed peristaltic pump is used to provide a continuous gas stream to the heated capillary inlet of the MS. The refrigerated air dryer is used to ensure that the air entering the 0-100 SLPM MFC is clean and free of condensable water vapor. The 0-100 SLPM MFC has its own Hewlett-Packard E3631A power supply, and is operated manually, independent of the Flow Control System and Tracer Injection System. The automated drain on the refrigerated air dryer has a 24 volt operated DC solenoid which can be switched on and off to allow purging of condensate.

3.1.4 Data Acquisition System

The data acquisition system is composed of two Keithley 2001 7 ½ digit multimeters, a Keithley 7002 scanner system, a Windows NT Personal Computer with monitor, and two Hewlett-Packard E3631A programmable power supplies. There is a GPIB IEEE488.2 compliant

interface card installed in the personal computer, which interfaces the computer to the aforementioned electronics.

3.1.5 Software

The main data acquisition PC is running Labview data collection software and uses the computer program `trace_inject.vi`. `Trace_inject.vi` uses the same subroutines as have been detailed in YMP-LBNL-TIP/TT-1.0, with a modified main routine which is responsible for controlling the injection tests. `Trace_inject.vi` is specifically designed to maintain a steady flow rate of ambient air, which will then be mixed for a finite amount of time with tracer gas. This is the configuration used for convergent tracer tests. The main routine `tracer_inject.vi` is included, as Attachment 3. `Trace_inject.vi` may need to be modified depending upon the desired testing boundary conditions required. For instance, if a slug injection of tracer is desired, followed by a fixed amount of ambient air, a routine will need to be added to control the ambient air MFC to inject the follow up mine air for a fixed amount of time. If `tracer_inject.vi` is used in a modified format, the modifications must be identified and documented in a scientific notebook and the new vi shall be saved using a unique identifier for permanent archiving. The Labview vis shall come under the control and documentation requirement of YMP-LBNL-QIP-SI.0, *Computer Software Qualification* and YMP-LBNL-QIP-SI.1, *Software Configuration Management*.

3.2 Calibration Requirements

3.2.1 MFC and DMM Calibration

The mass flow controllers and the digital multimeters are on a yearly calibration cycle. A staff member shall verify before commencing tracer testing that each instrument's calibration is still current. If any instrument is found not to be within calibration or the data it is generating are suspect, the equipment shall be removed from service and replaced with a calibrated unit. The unit requiring calibration shall be treated as per the requirements of YMP-LBNL-QIP-12.0.

3.2.2 Instrument Accuracy

The accepted accuracy in the field for the MFCs are $\pm 10\%$, although they are calibrated by the manufacturer to $\pm 1\%$. The Setra pressure transducers have a calibrated accuracy of $\pm 0.1\%$, but under this TIP they shall provide data with an accuracy of $\pm 0.25\%$.

3.2.3 Mass Spectrometer Calibration

During gas tracer testing, the Balzer's MS requires calibration in the field. For each tracer test it is required that a calibration be performed at least once before the test, during the test on a daily basis, and after the conclusion of the test. The calibration standards shall bracket the concentrations of tracer that are expected to be analyzed during gas tracer testing. The calibration of the Balzer's MS is covered under a separate TIP, YMP-LBNL-TIP/TT-3.0, *Calibration and Tuning of the Balzer's Mass Spectrometer for Tracer Test Usage in the Drift Scale Test Area Using Reference Calibration Bags*, and is not described in this document. The tuning of the MS is described in YMP-LBNL-TIP/TT-3.0.

3.3 Tracer Test Configuration

3.3.1 Test Geometry

Tracer testing can be performed with a wide variety of test geometries and boundary conditions. Several identified testing geometries are identified here, but this list is not meant to be exhaustive or limit future tracer test configurations. The injection gas stream is used to introduce tracer into the formation and the withdrawal gas stream is used for analysis of tracer arrival.

3.3.2 Test Types

Convergent tracer tests utilize a large withdrawal flow rate to draw tracer towards a central location for analysis, while a divergent test utilizes a large tracer injection flow rate to introduce tracer into a central test location where it becomes dispersed. Both convergent and divergent tests can be performed in monopole and dipole configurations. A monopole test implies that the flow field is set up such that there is a single large flow rate and either the injection (for convergent tests) or withdrawal (for divergent tests) is at a rate, which is sufficiently small such that it may be ignored during subsequent analysis. A dipole test utilizes simultaneous injection and withdrawal flows, which may be either equal in strength or unequal. Where there is a large disparity in injection and withdrawal flow rates the test is referred to as a weak dipole test.

3.3.3 Injection Methodology

Gas tracer injections can be continuous, which will create a constant concentration and flux boundary condition, or may be a pulse or slug. A pulse or slug injection indicates that gas tracer was injected for a shorter amount of time than the duration of the tracer test.

3.4 Test Preparation

Staff members preparing for gas tracer testing shall perform the actions described below.

3.4.1 Electronics

- A. Before turning on the electronics for testing, turn on the air conditioner on the Electronic Rack and verify that it is properly cooling the rack.
- B. Turn on the Keithley Instruments, the Hewlett-Packard power supplies, and the $\pm 15V$ power supply used for the MFCs, which are all located in the electronic enclosure.
- C. Turn on the computer monitor and finally the computer.
- D. Verify that the power supply to the Setra transducers, which is located in the Acoustic Emission Electronic Enclosure in the main data acquisition office, is turned on and set to 24 volts.

3.4.2 Flow Control Components

- A. Verify visually that the manually operated valves located on the injection and withdrawal lines are in the open position.
- B. Turn on the twin-tower desiccant dryer, verify that the ambient air supply valve open, and that the regulator on set the output of the desiccant dryer to 60 PSIG.
- C. Verify that the two manually operated 1/8" supply air line valves that are located near the pressure regulator are open.
- D. The inlet and outlet filters on the twin-tower dryer have visual gauges, which indicate the status of the filters. If at any time the

indicators appear red, replace the filters at the next convenient time. It is not necessary to interrupt ongoing tests to change a filter. This is anticipated to be a once a year or less frequent maintenance item.

- E. Verify that the regulator on the tracer gas injection cylinder is connected and the outlet pressure is adjusted to at least 60 PSIG.

3.4.3 Balzer's Mass Spectrometer

A Mass Spectrometer Startup

Turn on the Balzer's mass spectrometer and allow it to pump down for at least 24 hours prior to start of calibrated data collection. The inlet orifice need not be opened until an hour before actual testing. With the inlet orifice closed, the pressure within the spectrometers vacuum chamber should reach 1.0×10^{-7} mbar. If it does not a vacuum leak may exist and the vacuum fittings should be checked for tightness. Vacuum pressure data collected from time of turning on the mass spectrometer to the commencement of tracer testing shall be utilized as a diagnostic tool to ensure that the mass spectrometer pressure is reaching a steady state value which is low enough for testing (5.0×10^{-8} mbar or lower). This pressure may be monitored by using the mass spectrometer computer and running the program service.exe located in the QS421 subdirectory folder.

B. Capillary Tube Heating

Heat the capillary inlet tube with a set point above the boiling point of water. This will prevent the clogging of the capillary due to the formation of condensate. A recommended temperature is 120°C, which is set using the up and down arrows on the front panel of the mass spectrometer.

C. Starting Measurements

To make measurements using the mass spectrometer, exit the service.exe program and start the measure.exe program. From the QS421 file folder, double click on the measure.exe icon. Proceed to turn on the detector (SEM) and emission filament. After verifying that there is a filter on the inlet of the mass spectrometer capillary tube, open the inlet valve. Open the desired monitoring parameter set and choose versus time. The ion current versus cycle time screen will appear. Proceed to log data to a file.

E. Calibration

Perform a calibration of the mass spectrometer by following YMP-LBNL-TIP/TT-3.0.

3.5 Data Collection

Executing trace_inject.vi starts the execution of the tracer test. The mass spectrometer shall already be analyzing the withdrawal gas stream prior to the start of the main tracer test routine.

3.5.1 Data Acquisition System

The data acquisition system continuously collects pressure, temperature, and flow rate information during each tracer test. The data files that are created consist of both raw and converted pressure, temperature, and flow rate information. Each data file is given a unique name based on the date and time that it was started, as well as a suffix that indicates what type, and whether it includes raw or processed data. The suffixes are .pres or .pres_cal for pressure data files and .rtd or .rtd_cal for temperature data files. The files with suffixes that have _cal in them are engineered units while the files that do not have _cal are in unprocessed format.

3.5.2 Mass Spectrometer

The Balzer's Mass Spectrometer collects data into a binary file that has the suffix .mdc in its dedicated laptop computer. It is required, at a minimum of once per day, to convert the binary cycle data into ASCII format using the Dispsav.exe program. This serves as a means for backing up the data, as well as to produce a file which is convenient for visual observation and loading into Microsoft Excel or other data processing software.

3.5.3 Notebook Records

Staff members shall record the following information in their scientific notebook used during testing:

- A. Name(s) of testing personnel
- B. Date and time of test
- C. MFC serial numbers for tracer and withdrawal controllers
- D. Zone of gas injection and injection flow rate
- E. Zone of gas withdrawal and withdrawal flow rate
- F. Targeted tracer concentration and duration of injection. (The actual tracer concentration and duration are recorded in electronic format and shall be entered in the scientific notebook when the data are processed.)
- G. Data collected under TIP YMP-LBNL-TIP/TT-3.0 while calibrating the MS. This shall include the identifiers of data files

generated, calibration concentrations, calibration bag identifiers, and sample cycle numbers.

- H. Any unusual occurrences or testing interferences with DST activities.
- I. Time at which and method used to ensure the completeness and accuracy of the data has been established and the method by which the security of the data is maintained.

4. RECORDS

4.1 Lifetime

The electronic data generated from Gas Tracer Testing are stored in two locations, the laptop computer which is used to operate the Omnistar MS, and the Data Acquisition System PC. All electronic data shall be backed up at the conclusion of each tracer test. Prior to data submittal to the YMP Technical Database, the data files shall be reviewed for completeness and content. The MS calibration files shall be reviewed to ensure that the calibrations and mass balances are reasonable. A simple comparison of the best fit slope and intercept between each calibration should reveal only small shifts in the calibration over time. Less than 20% per day is acceptable with less than 10% being considered ideal. For long duration tracer testing (more than two days) it may be necessary to apply multiple calibrations to a single data set to ensure that the accuracy of converting ion currents to tracer concentration is kept to less than 20% drift. The data acquisition files shall be reviewed to ensure that flow rates are steady and within expected tolerances for each MFC.

The related data shall be turned over to the Technical Data Coordinator in accordance with YMP-LBNL-QIP-SIII.3, *Submitting Key Data to the Yucca Mountain Project Office*, for submittal to the Technical Database (TDB).

4.2 Non-Permanent

None

4.3 Controlled Documents

Technical Implementing Procedure

4.4 Records Center Documents

Records associated with this procedure shall be submitted to Records Processing Center (RPC) in accordance with AP-17.1Q, *Record Source Responsibility for Inclusionary Records*.

5. RESPONSIBILITIES

- 5.1 The **Project Manager** is responsible for final approval of the new, revised or modified TIP and for final approval of the rescission of the TIP.
- 5.2 The **EA Manager** is responsible for approving the new, revised or modified TIP, and for the rescission of the TIP.
- 5.3 The **OQA Representative** is responsible for reviewing and concurring with the TIP.
- 5.4 The **Principal Investigator (PI)** or designee is responsible for assuring full compliance with this procedure. The PI shall require that all personnel assigned to work to this procedure shall: 1) have the necessary qualifications and training to adequately perform the procedure; and 2) that they shall have a working knowledge of the LBNL QA Program.
- 5.5 **Staff Members** are responsible for following this procedure and turning over related documentation to the Records Coordinator for the submittal to the Records Processing Center in accordance with AP-17.1Q.

Special qualifications and/or training unique to the conduct of this procedure are as follows: 1) In the acquisition phase of the project, field supervisors and/or managers (or their designates) shall have a working knowledge of mechanical and electronic equipment; 2) Field personnel shall have all safety training as required by LBNL Environmental Health and Safety regulations to operate basic electrical and low pressure compressed gas systems; 3) field personnel shall be in compliance with ESF General Underground Safety Training requirements.

- 5.6 **Document Control Staff** are responsible for providing the controlled distribution of the TIP and modifications thereof

6. ACRONYMS AND DEFINITIONS

6.1 Acronyms

DST Drift Scale Test

LBNL Lawrence Berkeley National Laboratory

MFC Mass flow controller

MS Mass Spectrometer

PC	Personal Computer
PI	Principal Investigator
SCCM	Standard cubic centimeter per minute
SLPM	Standard liter per minute
TDB	Technical Database
TIP	Technical Implementation Procedure

6.2 Definitions

Calibration: The process of establishing the accuracy of a standard or measuring device, which may require resetting parameters on the device to improve its accuracy.

Staff Member: Any scientist, engineer, research or technical associate, technician, or student research assistant performing quality-affecting work for YMP-LBNL.

Technical Implementing Procedure: Each TIP describes YMP-LBNL technical tasks that (1) are repetitive, (2) are standardized, and (3) can return different results if deviation from the sequence of steps occur.

7. REFERENCE

AP-17.1Q, *Record Source Responsibility for Inclusionary Records*

YMP-LBNL-QIP-5.2, *Preparing Quality & Technical Implementing Procedures*

YMP-LBNL-QIP-12.0, *Control and Calibration of Measuring and Testing Equipment*

YMP-LBNL-QIP-SI.0, *Computer Software Qualification*

YMP-LBNL-QIP-SI.1, *Software Configuration Management*

YMP-LBNL-QIP-SIII.0, *Scientific Investigation*

YMP-LBNL-QIP-SIII.3, *Submitting Key Data to the Yucca Mountain Project Office*

YMP-LBNL-TIP/TT-1.0 *Drift Scale Test Constant Mass Flux Air-Permeability Testing in Hydrology Boreholes*

YMP-LBNL-TIP/TT-3.0 *Calibration and Tuning of the Balzer's Mass Spectrometer for Tracer Test Usage in the Drift Scale Test Area Using Reference Calibration Bags*

8. ATTACHMENTS

Attachment 1	Gas Flow Control System
Attachment 2	Gas Withdrawal System for Conducting Gas Tracer Tests in the DST Hydrology Boreholes
Attachment 3	Labview Software for Conducting Gas Tracer Tests in the DST Hydrology Boreholes

9. REVISION HISTORY

09/30/98 Revision 0, Modification 0:

This is the initial issue of this procedure.

10. Approvals

Preparer: Barry Freifeld

Date

Technical Reviewer: Nicholas Spycher

Date

Technical Reviewer: Peter Persoff

Date

EA Reviewer: Nancy Aden-Gleason

Date

OOA Concurrence: Stephen D. Harris

Date

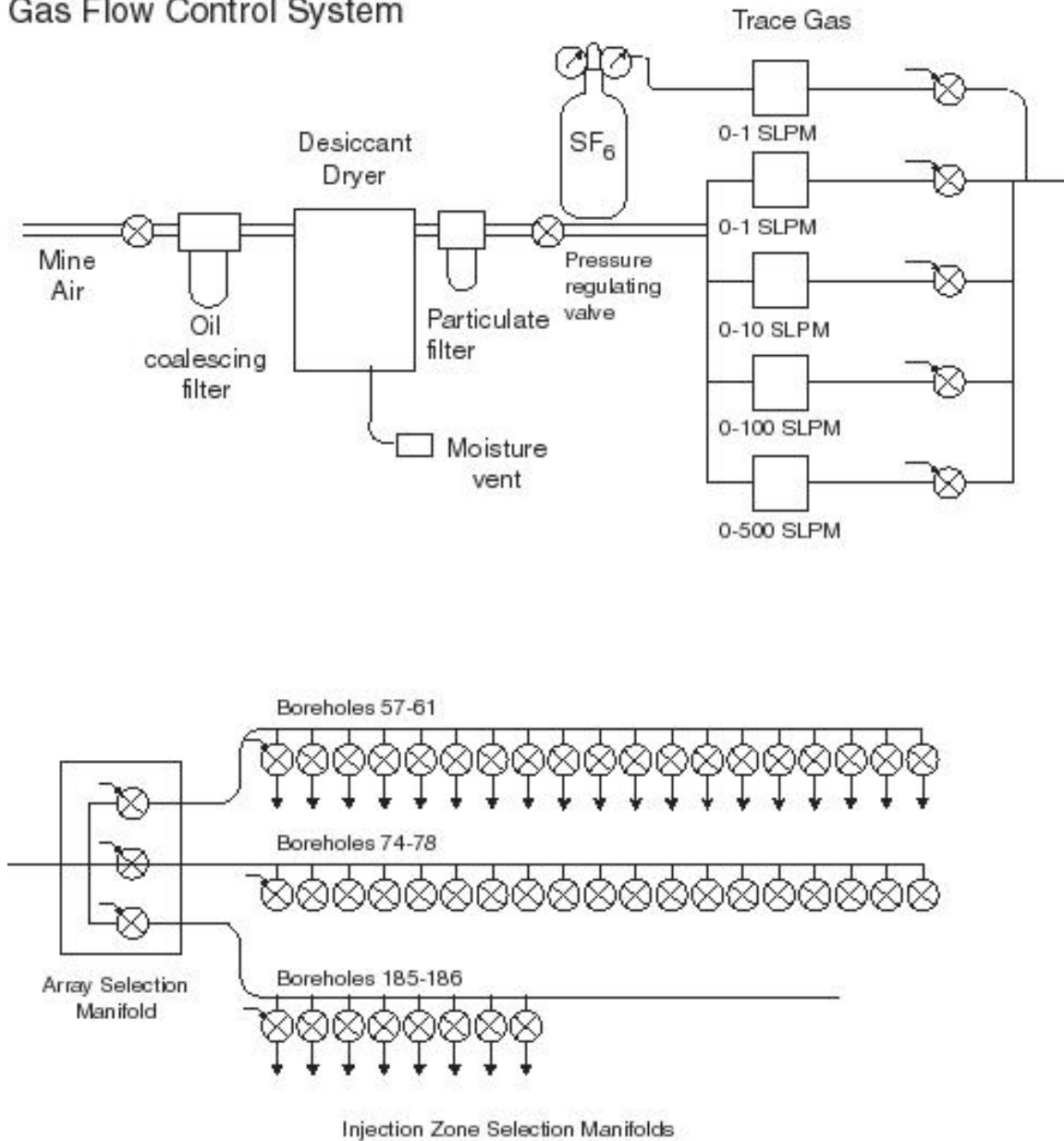
Principal Investigator: Yvonne W. Tsang

Date

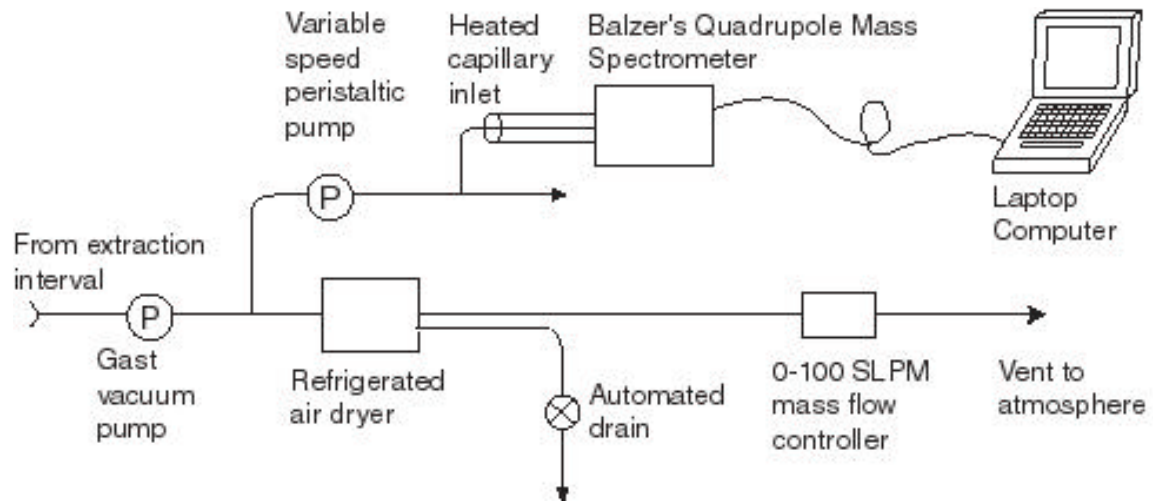
Project Manager: Gudmundur S. Bodvarsson

Date

Gas Flow Control System

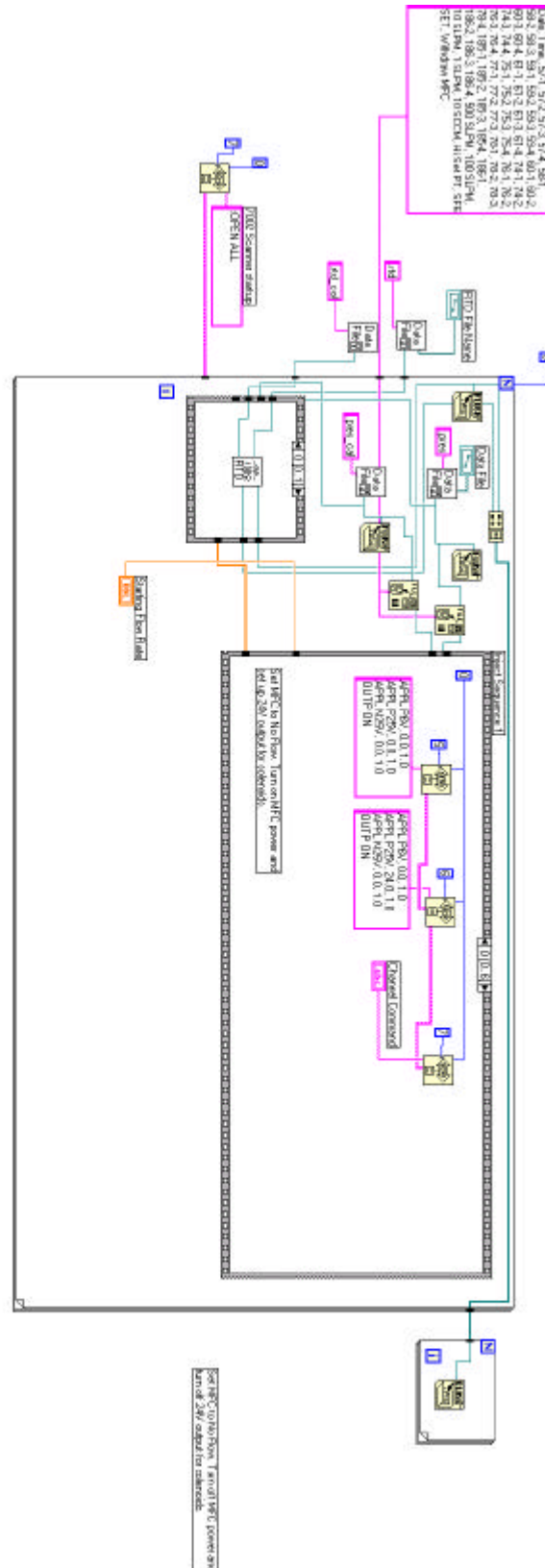


Injection flow control system for performing Gas Tracer Tests in the DST Hydrology Boreholes.

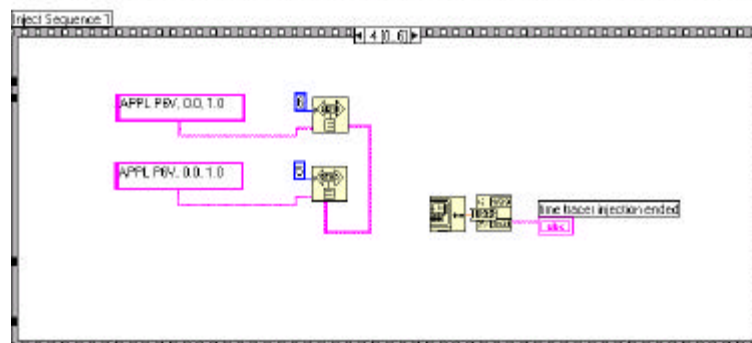
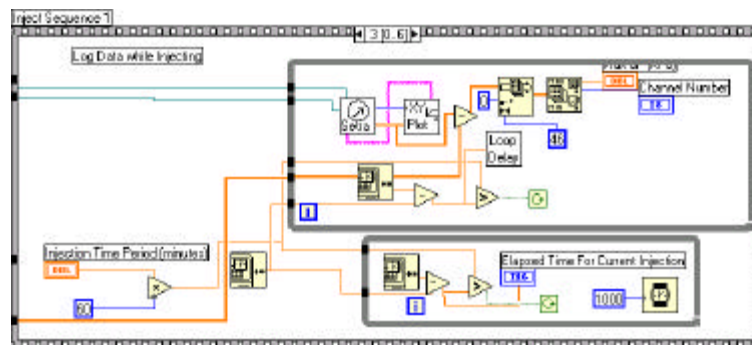
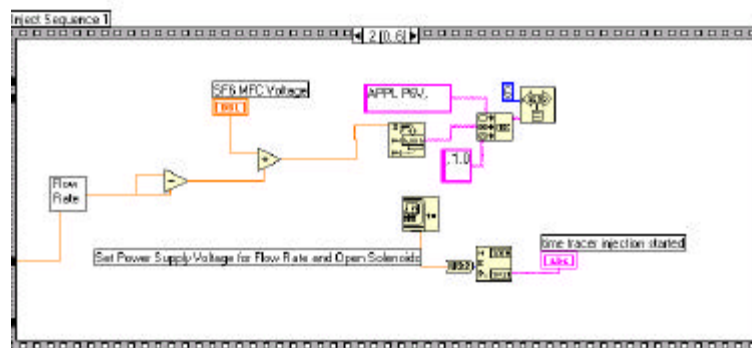
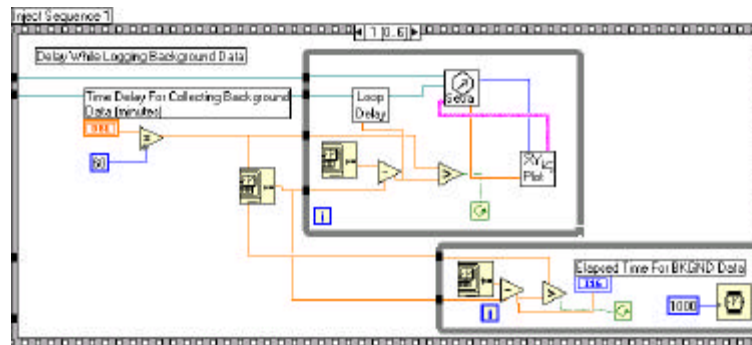


Gas Withdrawal System for conducting Gas Tracer Tests in the DST Hydrology Boreholes.

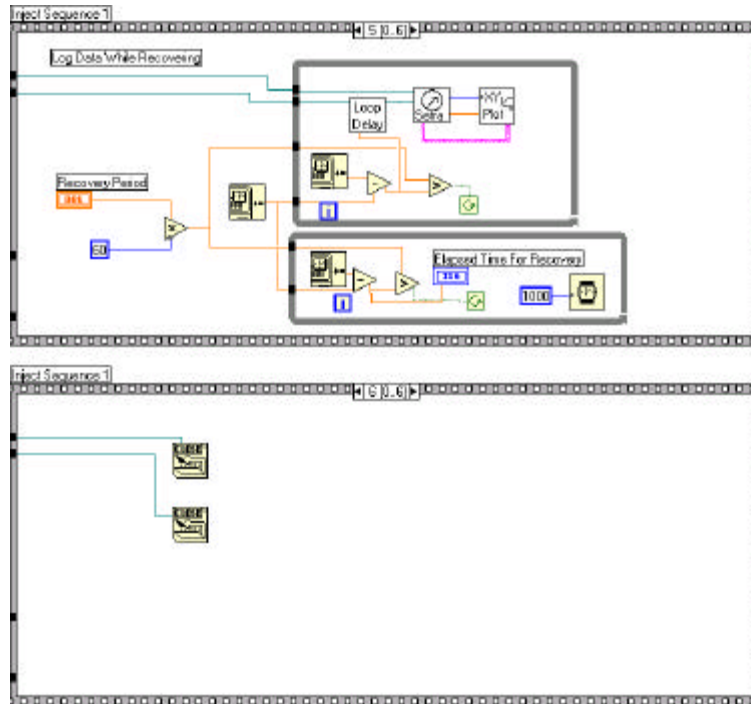
Labview Software for Conducting Gas Tracer Tests in the DST Hydrology Boreholes



Labview Software for Conducting Gas Tracer Tests in the DST Hydrology Boreholes



Labview Software for Conducting Gas Tracer Tests in the DST Hydrology Boreholes



RTD File Name &E:\41358 8-33 PM\rd Data File &E:\41358 8-33 PM\pres	Time Delay For Collecting Background Data (minutes) 5.00	Max dP (Psi) 0.1325
Starting Flow Rate 0.50 SPC MFC Voltage 1.00	Injection Time Period (minutes) 30.00 Recovery Period 6000.00	Channel Number 28 Time Tracer Injection ended Time Tracer Injection started
Channel Command CLOS (8/4/20) CLOS (8/12/20)	Elapsed Time For BKGND Data 25 Elapsed Time For Current Injection 122 Elapsed Time For Recovery 62	